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SPECIFICATION FOR LINSEED OIL, BOILED, FOR PAINTS

(Second Revision)

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Indian Standard

SPECIFICATION FOR LINSEED OIL, BOILED, FOR PAINTS

(Second Revision)

0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Indian Standards Institution on 25 March 1976, after the draft finalized by the Raw Materials for Paint Industry Sectional Committee had been approved by the Chemical Division Council.

0.2 Linseed oil, boiled (also known as double boiled, and pale boiled) is used in the manufacture of paints and varnishes, oil cloth, linoleum, printing inks, artificial rubber and also as a foundry core oil.

0.3 This standard was first published in 1950 and subsequently revised in 1968 amalgamating it with IS : 78-1950*. In this second revision an additional requirement for rosin (when rosinate driers are used) has been included on a specific request from Ministry of Defence.

0.4 This standard achieves alignment with JSS 1022 ' Specification for oil, linseed, boiled, ammunition ' issued by the Department of Standardization, Ministry of Defence, Government of India.

0.5 This standard contains clause **5.1** which calls for agreement between the purchaser and the supplier.

0.6 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960†. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes requirements and methods of sampling and test for linseed oil, boiled, for paints. Apart from paint industry and other allied applications, it is also used as foundry core oil.

*Specification for linseed oil, pale boiled, for paints. (Since withdrawn).
†Rules for rounding off numerical values (*revised*).

1.1.1 It does not cover the requirements of raw and refined linseed oil which are covered by IS : 75-1973*.

2. TERMINOLOGY

2.1 For the purpose of this standard, the definitions given under **2** of IS : 74-1966† shall apply.

3. TYPES

3.1 There shall be the following two types of material:

Type 1 — boiled, and
Type 2 — pale boiled.

4. REQUIREMENTS

4.1 Description — The material shall be prepared from genuine linseed oil obtained from *Linum usitatissinum* Linn., fam. Linaceae and necessary driers. It shall be clear and free from sediment and other insoluble matter.

4.2 Freedom from Fish, Hempseed, Castor and Cottonseed Oils — The material shall pass the test when tested as prescribed in Appendix A.

4.3 Lead-Free Material — When a lead-free material is required, it shall contain not more than 0·03 percent of lead or compounds of lead or mixture of both, calculated as metallic lead (Pb), when tested as prescribed under **22** of IS : 74-1966†.

4.4 Colour (for Type 2 Only) — The colour of the material, when examined through a $\frac{1}{4}$-in glass cell in a standard Lovibond tintometer, shall be not deeper than a combination of 10 Y + 2 R on the Lovibond scale.

4.5 Rosin Acid Content (When Rosinate Driers are Used) — The material, when intended for Defence use, shall contain rosin acid not more than 1·0 percent by mass when tested as prescribed in Appendix B.

4.6 The material shall also comply with the requirements given in Table 1.

5. PACKING AND MARKING

5.1 Packing — The material shall be supplied packed in suitable containers as agreed to between the purchaser and the supplier.

5.2 Marking — The containers shall be marked with the following particulars:

a) Name and type of the material;
b) Manufacturer's name and his recognized trade-mark, if any;
c) Net mass of the material in the container;
d) Batch No. or Lot No. in code or otherwise; and
e) Month and year of manufacture.

*Specification for linseed oil, raw and refined (*second revision*).
†Methods of sampling and test for drying oils for paints (*first revision*).

TABLE 1 REQUIREMENTS FOR LINSEED OIL, BOILED, FOR PAINTS

(*Clause* 4.6)

Sl No.	Characteristic	Requirement for		Method of Test (Ref to Cl No. in IS : 74-1966*)
		Type 1	Type 2	
(1)	(2)	(3)	(4)	(5)
i)	Relative density at 30°C/30°C	0·931 to 0·945	0·929 to 0·943	6
ii)	Acid value, *Max*	8	6	9
iii)	Saponification value	190 to 196	190 to 198	10
iv)	Unsaponifiable matter, percent by mass, *Max*	2·5	2·0	12
v)	Drying time, *Max*	18 hours	18 hours	15
vi)	Ash content, percent by mass, *Max*	0·5	0·3	19

*Methods of sampling and test for drying oils for paints (*first revision*).

5.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

6. SAMPLING

6.1 Representative samples of the material shall be drawn as prescribed under 3 of IS : 74-1966*.

7. TEST METHODS

7.1 Tests shall be conducted as prescribed in IS : 74-1966* and Appendices A and B. References to relevant clauses of IS : 74-1966* are given in **4.3,** and col 5 of Table 1.

7.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960†) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Methods of sampling and test for drying oils for paints (*first revision*).
†Specification for water, distilled quality (*revised*).

APPENDIX A

(*Clause* 4.2)

TEST FOR FREEDOM FROM FISH, HEMPSEED, CASTOR AND COTTONSEED OILS

A-1. FREEDOM FROM FISH AND HEMPSEED OILS

A-1.0 Outline of the Method — The addition of bromine to unsaturated fatty acids of fish and hempseed oils results in a petroleum hydrocarbon solvent insoluble bromide whereas that formed with linseed oil fatty acids is soluble in petroleum hydrocarbon solvent. This principle is used in the test for freedom from fish and hempseed oils. Bromine is added to the material dissolved in petroleum hydrocarbon solvent at low temperature and solution examined for any cloudiness developed.

A-1.1 Reagents

A-1.1.1 *Nitric Acid-Potassium Nitrate Solution* — 10 percent (*m/v*) solution of nitric acid saturated with potassium nitrate.

A-1.1.2 *Petroleum Hydrocarbon Solvent* — 145/205 (low aromatic) (conforming to IS : 1745-1966*).

A-1.1.3 *Bromine*

A-1.2 Procedure

A-1.2.1 Shake 1 to 2 ml of the oil with a 10 percent solution of nitric acid saturated with potassium nitrate. If the oil and acid solution are warmed prior to shaking, the separation will be immediate. Separate the oil and remove the traces of moisture by filtering the oil through a double layer filter paper. Dissolve the clear oil in 20 ml of petroleum hydrocarbon solvent in a test tube or a small flask. Mix the solution thoroughly and place the container in freezing mixture having a temperature of $-10 \pm 1{\cdot}0°C$. Add bromine drop by drop from a burette avoiding vigorous action until a considerable excess is present as indicated by the presence of a strong red colour. Shake the container well and then allow it to stand in cold water for 15 minutes.

A-1.2.1.1 The material shall be taken to have passed this test if no cloudiness develops.

A-2. FREEDOM FROM CASTOR OIL

A-2.0 Outline of the Method — The presence of castor oil develops turbidity when acidified petroleum hydrocarbon solution is treated with ammonium molybdate solution.

*Specification for petroleum hydrocarbon solvents (*first revision*).

A-2.1 Reagents

A-2.1.1 *Acidified Petroleum Hydrocarbon Solvent* — Add 2 ml of hydrochloric acid (relative density 1·19) to 100 ml of petroleum hydrocarbon solvent 145/205 (low aromatic) (conforming to IS : 1745-1966*).

A-2.1.2 *Molybdate Reagent* — Dissolve 1·25 g of ammonium molybdate in 100 ml of sulphuric abid (relative density 1·84; conforming to IS : 266-1961†).

A-2.2 Procedure — Take about one millilitre of the material in a clean dry test tube and add 10 ml of acidified petroleum hydrocarbon solvent. Shake vigorously for 2 minutes and add a drop of molybdate reagent. The development of turbidity indicates the presence of castor oil.

A-3. FREEDOM FROM COTTONSEED OIL

A-3.0 Outline of the Method — Development of red colour on heating the oil with a solution of sulphur in carbon disulphide indicates the presence of cottonseed oil. The test is also responded by hempseed oil.

A-3.1 Reagent

A-3.1.1 *Sulphur Solution* — Prepare one percent (*m/v*) solution of sulphur in carbon disulphide and add an equal volume of amyl alcohol.

A-3.2 Procedure — Take about 5 ml of the material and add an equal volume of sulphur solution. Mix thoroughly by shaking and heat gently on a water-bath for a few minutes with occasional shaking until the carbon disulphide has boiled off and the sample stops foaming. Place the tube in an oil bath maintained at 110 to 115°C and hold for one or two hours. Development of red colour at the end of this period indicates the presence of cottonseed oil.

APPENDIX B

(*Clause* 4.5)

DETERMINATION OF ROSIN ACIDS

B-1. OUTLINE OF THE METHOD

B-1.1 A known quantity of the material is saponified with caustic alkali and hydrolysed with hydrochloric acid. The rosin acids and fatty acids thus obtained are extracted with benzene, washed and selective esterification of fatty acids is carried out in presence of rosin acids.

*Specification for petroleum hydrocarbon solvents (*first revision*).
†Specification for sulphuric acid (*revised*).

B-2. REAGENTS

B-2.1 Alcoholic Potassium Hydroxide Solution — 10 percent.

B-2.2 Hydrochloric Acid — relative density 1·19.

B-2.3 Benzene

B-2.4 Acetone

B-2.5 Esterification Solution — Mix 500 ml of *n*-butanol, 500 ml of benzene, and 6 g of concentrated sulphuric acid. Place the mixture in a flask, attach a moisture trap and reflux for 30 minutes to form butylsulphuric acid. Cool and store in a glass-stoppered bottle.

B-2.6 Alcoholic Potassium Hydroxide Solution — 0·2 to 0·25 N, accurately standardized.

B-2.7 Indicator — 0·05 percent phenolphthalein or thymol blue indicator.

B-3. PROCEDURE

B-3.1 To the weighed sample in 500-ml conical flask add 50 ml of alcoholic potassium hydroxide, attach to a suitable condenser and reflux on hot water-bath for 1 hour. After refluxing, remove the flask from the hot water-bath and cool to room temperature under tap water. Add 100 ml of water, then add 20 to 25 ml of hydrochloric acid. The solution shall be distinctly acidic. Reflux for 5 minutes again and cool.

B-3.2 Transfer the sample quantitatively to a one-litre separatory funnel, wash the conical flask with 50 ml of water, 25 ml of benzene, 50 ml of water and 15 ml of acetone respectively and add to the funnel. Shake, allow the layers to separate and draw off the lower aqueous layer into a second one-litre separatory funnel. Extract the aqueous layer with a second 50-ml portion of benzene and drain the aqueous layer into a third one-litre funnel. Add the second benzene extract to the first. Repeat the extraction of the aqueous layer with successive 50-ml portions of benzene (total number not less than 3). Discard the water layer. Combine the benzene extracts and wash with three 50-ml portions of water or until wash water is free from acid. Transfer the washed benzene extract to a weighed 250-ml conical flask with the aid of 25 ml of benzene. Evaporate the benzene on the steam-bath. Add 5 g of anhydrous ethyl alcohol to remove any water present by azeotropic distillation, cool and weigh.

B-3.3 Weigh a portion of the benzene extract (**B-3.2**) accurately into a 250-ml conical flask with ground joint according to the following:

Rosin Acids, percent	*Sample Size*, g
(1)	(2)
0 – 5	5 – 8
5 – 20	3 – 5
20 – 50	2 – 3
50 – 100	1 – 2

Using a burette or constant delivery pipette, accurately measure 50 ml of esterification solution into the flask and add a few boiling stones. Attach a moisture trap and condenser, place on a hot plate, heat to boiling and reflux for 20 minutes. The boiling shall be vigorous at all times, so that the water which is formed separates rapidly. At the end of heating period allow the flask and contents to cool somewhat. Remove the flask, cool immediately to room temperature, and titrate with alcoholic potassium hydroxide solution. Make a blank titration on the same volume of esterification solution after refluxing it in the same manner.

NOTE — Light coloured samples may be titrated with phenolphthelein or thymol blue indicator. Dark samples and those containing small amounts of mineral acids or alkalies are preferably titrated potentiometrically.

B-4. CALCULATION

$$\text{Rosin acids (as abietic acid), percent by mass} = \frac{(A - B) \times N \times 30{\cdot}78}{M} - 0{\cdot}3$$

where

A = volume in ml of alcoholic potassium hydroxide solution required for the sample,

B = volume in ml of alcoholic potassium hydroxide solution required for the blank,

N = normality of alcoholic potassium hydroxide solution,

M = mass in g of the material taken for the test, and

0·3 = experimentally determined constant to correct for unesterified fatty acids.

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